Oklahoma State University
Department of Chemistry

Service Facilities for Nanoscience

Oklahoma State University has excellent characterization facilities for solution processed building blocks of nanostructured materials and full time Ph.D. staff to provide service for outside users. The facilities are currently used by researchers throughout the State of Oklahoma. We would like them to be a part of the National Nanotechnology Infrastructure Network.

Warren T. Ford, Regents Professor
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Oklahoma Statewide Shared NMR Facility
Oklahoma State University
Department of Chemistry

Established 1996 with grants from

- **National Science Foundation**
- **Oklahoma State Regents for Higher Education**
- **W. M. Keck Foundation**
- **Conoco, Inc.**
- **S. R. Noble Foundation**

Warren T. Ford, Faculty Director 1996-2002
K. Darrell Berlin, Faculty Director 2002-

Staff
Margaret A. Eastman, Ph.D., Senior Spectroscopist
Facility Manager, Ph.D., position currently vacant

The NMR Facility serves the State of Oklahoma as well as Oklahoma State University. The most frequent outside users are from

- **University of Oklahoma**
- **Samuel Roberts Noble Foundation**

Oklahoma Statewide Shared NMR Facility
Equipment List

Varian UNITY INOVA 600 MHz Spectrometer

Installed 1997
14.1 T 51-mm Bore Oxford Superconducting Magnet
Ultra·nmr 40 Channel Shims
Four RF Channels
Four linear amplifiers (AMT): 300W, 10-245 MHz; two 300W, 6-220 MHz; 100W, 550-640 MHz ($^{1}H/^{19}F$)
Three Waveform Generators
Performa II Pulse Field Gradient Amplifier (X, Y, Z axes)
Deuterium Decoupling
Temperature Controller

Probes for UNITY INOVA 600

Nalorac 5-mm ^{1}H {^{13}C,^{15}N} Z PFG triple resonance ID Probe, VT (-15 to 55 °C)
Nalorac 3-mm ^{1}H {^{13}C,^{15}N} Z PFG triple resonance ID Probe, VT (-15 to 55 °C)
Nalorac 5-mm ^{1}H {^{15}N-^{31}P } Z PFG ID Probe, VT (-50 to 130 °C)
Varian 5-mm ^{15}N-^{31}P {^{1}H} Broadband Probe, VT (-150 to 200 °C)
Varian 4-mm ^{13}C {^{1}H} Nano·nmr Probe, VT (-5 to 50 °C)

Varian UNITY INOVA 400 MHz Spectrometer

Installed 1996 (magnet 1989)
9.4 T 54-mm Bore Oxford Superconducting Magnet
Enhanced 23 Channel Shims
Two RF Channels.
Two linear amplifiers (AMT): 300W, 6-220 MHz; 100W, 200-500 MHz ($^{1}H/^{19}F$)
Two Waveform Generators
Pulsed Field Gradient Amplifier (Z axis)
Temperature Controller

Probes for UNITY INOVA 400

Nalorac 5-mm ^{1}H {^{15}N-^{31}P} Z PFG Indirect Detection Probe, VT (-20 to 60 °C)
Varian 5-mm ^{1}H -$^{19}F/^{15}N$-^{31}P Switchable Probe, VT (-150 to 200 °C)
Varian 5-mm ^{1}H /$^{19}F/^{13}C/^{31}P$ Auto·nmr Probe, VT (-150 to 200 °C)
Varian 5-mm ^{15}N-^{31}P {^{1}H} solids MAS Probe VT (-125 to 125 °C)

Varian GEMINI 2000 300 MHz Spectrometer

Installed 1997
7.05 T 54-mm Bore Oxford Superconducting Magnet
13 Channel Shims
Two RF channels
Two linear amplifiers (AMT): 150W, 30-122 MHz; 50W, 282-300 MHz ($^{1}H/^{19}F$)
Temperature Controller

Probe for GEMINI 300

Varian 5-mm $^{1}H/^{19}F/^{13}C/^{31}P$ Auto·nmr Probe, VT (-150 to 200 °C)

Chemagnetics CMX-II 300 MHz Solid-State Spectrometer

Installed 1993
7.07 T 89-mm Bore Superconducting Magnet
20 Channel Shims
Three RF channels
Three amplifiers: 300W ^{1}H (Amplifier Systems); 1kW, 10-86 MHz (ENI); 1 kW, 6-220 MHz (AMT)
Temperature Controller
Magic-Angle Spinning Speed Controller (+/- 5 Hz)

Probes for CMX-II 300

(All probes except the DOR are equipped for variable temperature operation at -150 to +250 °C.)

Double resonance ^{15}N-$^{31}P\{^{1}H\}$ 7.5 mm 0-7 kHz MAS Probe
Double resonance ^{15}N-$^{31}P\{^{1}H\}$ 5 mm 0-12 kHz MAS Probe with low carbon background
Double resonance ^{73}Ge-$^{31}P\{^{1}H\}$ 7.5 mm 0-7 kHz MAS Probe with low Al background
Triple resonance (^{1}H-X-Y) 5 mm MAS Probe, 17.2-122 MHz observe with low Al background
Double rotation (DOR) Probe (quadrupolar nuclei)
Wideline ^{2}H Probe

RESUME

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Oklahoma State University
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EDUCATION

University of California, Santa Barbara	B.A.	1979	Biochemistry, *With Highest Honors*
University of California, Santa Barbara	M.A.	1980	Biochemistry (no thesis)
University of North Carolina, Chapel Hill	Ph.D.	1986	Physical Chemistry

APPOINTMENTS

1986—1988	Postdoctoral Fellow, Department of Chemistry, Cornell University, Ithaca, New York
1989--1991	National Institutes of Health NRSA Postdoctoral Fellow, Department of Chemistry, University of California, Berkeley
1991—1992	Visiting Assistant Professor of Chemistry, Colby College, Waterville, Maine
1992—1993	Visiting Assistant Professor of Chemistry, Bucknell University, Lewisburg, Pennsylvania
1994—1995	Visiting Assistant Professor of Chemistry, Clark University, Worcester, Massachusetts
1995- present	Senior Spectroscopist, Oklahoma Statewide Shared NMR Facility, Oklahoma State University, Stillwater

RESEARCH INTERESTS

Applications of NMR to study structure and dynamics in a variety of systems including soil components, nanoparticles, sol-gel materials, inorganic/organic composites, polymers, proteins, and glasses. Current projects include dynamics of benzene in humic and fulvic acids using solid-state deuterium NMR, and protein structure determination in the liquid-state.

SELECTED PUBLICATIONS

S. Liu, U. Fooken, C. M. Burba, M. A. Eastman, and R. J. Wehmschulte, 2003. "Synthesis and Characterization of Amorphous Nanostructured HalO", submitted to *Chem. Mater*.

♦ G. A. Caignan, R. Deshmukh, A. Wilks, Y. Zeng, H. Huang, P. Moenne-Locoz, R. A. Bunce, M. Eastman, and M. E. Rivera, 2002. "Oxidation of Heme to β- and δ-Biliverdin by *Pseudomonas aeruginosa* Heme Oxygenase as a Consequence of an Unusual Seating of the Heme", *J. Am. Chem. Soc*., 124(50), 14879-14892.

Z. N. Utegulov, M. A. Eastman, S. Prabakar, K. T. Mueller, A. Y. Hamad, J. P. Wicksted, and G. S. Dixon, 2002. "Structural Characterization of Eu_2O_3-MgO-Na_2O-Al_2O_3-SiO_2 Glasses with Varying Eu_2O_3 Content: Raman and NMR Studies", *Journal of Noncrystalline Solids* 315(1-2), 43-53.

D. K. Nagesha, X. Liang, A. A. Mamedov, G. Gainer, M. A. Eastman, M. Giersig, J.-J. Song, T. Ni, and N. A. Kotov, 2001. "In2S3 Nanocolloids with Excitonic Emission: In_2S_3 vs CdS Comparative Study of Optical and Structural Characteristics", *J. Phys. Chem. B*, 105(31),

7490-7498.
S. Niwayama, Y. Inouye, and M. Eastman, 1999. "Solid-state NMR and X-ray diffractional analysis of conformational effects in _-symmetric bicyclo[2.2.1]hept-2-ene diester and monesters", *Tetrahedron Lett.*, 40, 5961-5965.
M. A. Eastman, 1999. "Examples of Hartmann-Hahn Match Conditions for CP/MAS between Two Half-Integer Quadrupolar Nuclei", *J. Mag. Reson.*, 139, 98-108.

COLLABORATORS & OTHER AFFILIATIONS

i. Collaborators

Allen Apblett, Oklahoma State University, Dept. of Chemistry
Kathleen E. Duncan, University of Oklahoma, Dept. of Botany and Microbiology
Jose de la Fuente, Oklahoma State University, Dept. of Veterinary Pathobiology
Nicholas A. Kotov, Oklahoma State University, Dept. of Chemistry
Karl T. Mueller, Pennsylvania State University, Dept. of Chemistry
Mark A. Nanny, University of Oklahoma, Dept. of Civil Eng. and Environ. Sci.
Mario E. Rivera, Oklahoma State University, Dept. of Chemistry
Rudolf J. Wehmschulte, University of Oklahoma, Dept. of Chemistry
James P. Wicksted, Oklahoma State University, Dept. of Physics

ii. Graduate and postdoctoral advisors

Lee G. Pedersen, Professor, University of North Carolina, Department of Chemistry, Chapel Hill, NC.
Harold A. Scheraga, Professor, Cornell University, Department of Chemistry, Ithaca, NY.
Alexander Pines, Professor, University of California, Department of Chemistry, Berkeley, CA.

iii. Thesis Advisees

None – Current position does not involve advising graduate students.

CURRENT SUPPORT:

"Bioavailability of Aromatic Hydrocarbons and Dynamics of Their Interactions with Natural Organic Matter: Linking Molecular- and Microbial-Scale Interactions" Co-Principal Investigator with Mark A. Nanny and Kathleen E. Duncan of the University of Oklahoma, NSF, 8-15-02 to 8-14-06, $1,100,000

NanoNet Microscopy Laboratory
Oklahoma State University
Department of Chemistry

Established 2002 with grants from

- **National Science Foundation, EPSCOR Research Infrastructure Improvement program**
- **Oklahoma State Regents for Higher Education**

Warren T. Ford, Faculty Director
Susheng Tan, Ph.D., Manager

The NanoNet Microscopy Laboratory serves researchers in academia and industry throughout the State of Oklahoma.

Oklahoma State University/NanoNet Microscopy Laboratory

DI Nanoscope IIIa Scanning Probe Microscopy Experimental Station

Installed October 2002

The DI Nanoscope IIIa MultiMode experimental station came with a Quadrex PhaseImaging Module for phase detection and breakout box for signal access in custom experiments. This station is also equipped with an Applications Module Head which enables Conductivity detection. It operates in quasistatic (contact-mode) and dynamic force microscopy (AFM/SFM), plus STM. The station performs a full range of SPM techniques for surface characterization. Special imaging capabilities include:

- Friction (lateral) force (LFM)
- Force modulation (FM)
- Intermittent contact ("tapping") / non-contact with phase measurement
- Electrostatic force microscopy (EMF)
- Magnetic force gradient (MFM)
- Conductivity (C-AFM)
- "Force volume" imaging for spatially resolved force spectroscopy (distance dependence) at low pixel density and acquisition speed (to characterize adhesion, stiffness and dynamic interaction regime, e.g. non-contact versus intermittent contact) is functional
- Fluid cell

Currently the MultiMode SPM is used for surface characterization of film formation and transformation of polymer latex nanoparticles, for conductivity/resistance characterization of semi-conductive nanoparticles and nanowires, and conformation identification of RNA on micro-arrays.

Witec AlphaSNOM Scanning Near-field Optical Microscope

Installation scheduled March 2003

The design of the Alpha-SNOM features a Confocal Microscope (CM), a Scanning Near-Field Optical Microscope (SNOM) and an Atomic Force Microscope (AFM) in one single instrument.

The AlphaSNOM uses an ultra precise capacitively controlled piezo platform to scan the sample in all three directions. The stage is used not only in SNOM and AFM, but also in confocal mode. Therefore it is possible to analyze the same sample area with different techniques and combine the benefits of all three modes.

- SNOM modes: Transmission, Fluorescence, Collection
- Confocal Microscopy modes: Transmission, Reflection, Fluorescence
- AFM: Contact force mode & Pulsed Force Mode
- Applications:
    - Polymer materials
    - Biological and biomedical materials
    - Liquid crystal materials
    - Latex
    - Semiconductors, etc.

Leica microsystems DMIRB Optical Microscope

Installed January 2003

The Leica DMIRB is a top-of-the range Inverted Research Microscope. It features:

- All stands with built in Bertrand lens and side port (80% / 100 %) and 12 Volt/100 Watt transmitted light illumination

- Sophisticated and outstanding optical performance of the new HC infinity objectives with long working distances; 5 objective lenses: 10x – 63x
- Optimized fluorescence, with centrable field and aperture diaphragms, ergonomic alignment and up to 4 rotatable filter blocks (Zero Pixel Shift)
- High variety of lamp houses with 6 lenses, mercury short arc photo optic lamp HBO 103W
- Most flexible Condenser system for Brightfield, Phase Contrast, Modulation Contrast, Dark Field Contrast, Polarization Contrast or Differential Interference Contrast, every condenser disc with 6 positions for prisms, stops or slits
- Field of View : 22 mm
- Camera ports: Side port, Front port, and Bottom port
- Camera: MediaCybernetics CoolSNAP-Pro Integrated digital monochrome camera with Image-Pro Plus 4.5 analytical imaging software
- Most flexible stage system with fixed stages

Malvern Instruments HPP5001 High Performance Particle Sizer

Installation scheduled February 2003

The Malvern HPPS 5001 High Performance Particle Sizer is a unique instrument capable of measuring the size of molecules in solution as well as the size of dispersions and emulsions and up to 20 volume% with patented Non-Invasive Back-Scatter (NIBS), from sub nanometer sizes to a few microns using dynamic light scattering. It is capable to measure:

- Size range: 0.6nm to 6 microns
- High sensitivity: Measure Lysozyme at 0.1 mg/ml
- Emulsions and dispersions up to 20 vol% with NIBS
- Range of sample types:
    - Polymers, proteins and other macromolecules in solution
    - Colloidal size materials from micelles to lattices, vesicles
    - Nanoceramics
    - Absorbing samples such as pigments and inks
- In standard cuvettes to minimize cross contamination
- Temperature range: 10°C to 90°C

Biographical Sketch

Susheng Tan
Manager of NanoNet Microscopy Lab
Oklahoma State University

Education: Jiangxi Normal University, Nanchang, China, B.Sc. 1987
Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, M.Sc. 1994 (with D. Zhang)
Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Ph.D. 1999 (with E. Zhou)
University of Minnesota, Postdoctoral Associate, 1999-2002 (with W. L. Gladfelter and G. D. Haugstad)

Appointments

Manager, Chemistry, Oklahoma State University, 2002-present
Research Assistant, Polymer Chemistry and Physics, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, July 1994-Sep. 1999
Lecturer of Chemistry, Ganzhou District School of Agriculture, Jiangxi, China, 1987-91

Awards and Honors

Excellence Prize of the President Scholarship, Chinese Academy of Sciences, 1999
Special Scholarship, Tokyo Institute of Polytechnics, Japan, 1998
Distinguished Student, Jiangxi Normal University, China, 1984/1985/1986/1987

Publications most related to nanotechnologies

Haugstad, G.; Dykstra, C.; Schmidt, R. H.; Hammerschmidt, J. A.; Staarup, D.; Tan, S.; Gladfelter, W. L.; Macosko, C. W.; and Cole P., AFM-Based methodologies for interfacial nanorheology and Nanotribology, *74th Annual Meeting, the Society of Rheology* (Oct 2002).

Haugstad, G.; Tan, S.; Staarup, D.; Gladfelter, W. L.; Avery, A., Approach-retract cycles with an oscillating nanotip: powerful methods to interrogate subsurface morphology and viscoelasticity on polymers, *224th ACS National Meeting, Boston, MA* (August 2002)

Tan, S.; Haugstad, G.; Gladfelter W. L.; and Andrew, A., Nanotribological behavior of thin poly(dimethylsiloxane) films, *223rd ACS National Meeting, Orlando, FL,* (April 2002)

Other selected publications

Yang, X.; Kong, X.; Tan, S.; Li G.; Zhou, E., Influence of temperature on lattice spacing of melt-crystallized poly(iminosebacoyliminodecamethylene), *Polym. Inter.* **2001**, *50*, 817-821

Yang, X.; Tan, S.; Li, G.; Zhou, E., the Dependence of Brill Transition on the Crystal Size of Nylon 10 10, *Macromolecules* **2001**, *34*, 5936-5942

Yang, X.; Tan, S.; Li, G.; Zhou, E., Lamellar Single Crystals of Nylon-10, 10 Grown from Dimethylformamide Solution, *J. Polym. Sci.: Part B: Polym. Phys.* **2001**, *39*(4), 729–735

Tan, S.; Su, A.; Yang, X.; Zhou, E., Crystallization and melting behavior of amorphous poly(iminosebacoyl iminodecamethylene), *J. Appl. Polym. Sci.* **2000**, *77*(5), 993-1002

Tan, S.; Su, A.; Li, W.; Zhou, E., New insight into the melting and crystallization behavior in semicrystalline poly(ethylene terephthalate), *J. Polym. Sci.: Polym. Phys.* **2000**, *38*(1), 53-60

Tan, S.; Su, A.; Luo, J.; Zhou, E.; Cheng, S. Z. D., Effect of physical aging on the microstructure and the crystallization of amorphous PEEKK, *Macromol. Chem. Phys.* **1999**, *200*, 2487-2493

Tan, S.; Su, A.; Luo, J.; Zhou, E., Crystallization kinetics of poly(ether ether ketone) (PEEK) from its metastable melt, *Polymer* **1999**, *40*(5), 1223-1231

Collaborative research

Nanotribological behavior of polymer fluids under confinement
with G. D. Haugstad, University of Minnesota, and A. Avery, Unilever Research, UK

Ph.D. Advisor: E. Zhou, Chinese Academy of Sciences
Postdoctoral Advisor: W. L. Gladfelter, University of Minnesota

Biographical Sketch

Warren T. Ford
Regents Professor of Chemistry
Oklahoma State University

Education: Wabash College, Chemistry, A.B. 1963, Cum Laude, Phi Beta Kappa
University of California, Los Angeles, Chemistry, Ph.D., 1967 (with D. J. Cram)
Harvard University, NSF Postdoctoral Fellow, 1967-8 (with P. D. Bartlett)

Appointments

Assistant Professor to Regents Professor, Chemistry, Oklahoma State University, 1978-present
Visiting Professor, Chemistry, UCLA, Jan.-May 2000
Visiting Professor, Organic Chemistry, Univ. of Nijmegen, The Netherlands, Aug.-Dec. 1992
Visiting Professor and Fulbright Fellow, Max-Planck-Institute for Polymer Research, Mainz, Germany, Aug. 1985 – May 1986
Senior Research Chemist, Rohm & Haas Co., Spring House, PA, 1975-78
Assistant Professor of Chemistry, Univ. of Illinois at Urbana-Champaign, 1968-75

Awards and Honors

Fellow of the American Association for the Advancement of Science, 1989
Oklahoma Scientist of the Year (Oklahoma Academy of Sciences), 1996
Oklahoma Chemist of the Year (American Chemical Society), 1999

Synergistic Activities

Program Co-chair, American Chemical Society Division of Polymer Chemistry (POLY) with responsibilities for planning future symposia and for scheduling the final program at one National Meeting per year, 1998-2000; organizer of several symposia at ACS meetings. Co-organizer of Polymers and Organic Chemistry 2002, a biannual international conference.

Project Coordinator for NanoNet (Oklahoma Network for Nanostructured Materials), one half of the science in the Oklahoma grant from the NSF EPSCoR Infrastructure Improvement Program, starting 02-01-02. NanoNet will provide new equipment for shared instrumentation facilities, seed grants for new investigators, graduate student stipends, postdoctoral stipends for interdisciplinary research, and workshops and short courses in nanoscale science and engineering at Oklahoma State University, the University of Oklahoma, and the University of Tulsa.

Current research group: 6 Ph.D. students, 2 postdoctoral associates

Publications most related to this proposal

Guldi, D. M.; Ford, W. T.; Nishioka, T., Rate constants of reactions with 2-cyano-2-propyl radical and triplet state lifetimes of low molar mass and polymeric substituted [60]fullerenes, *Electrochem. Soc. Proc.* **1999**, *99-12*, 315-318.

Ford, W. T.; Nishioka, T.; Qiu, F.; D'Souza, E.; Choi, J.-p.; Kutner, W.; Noworyta, K., Structure Determination and Electrochemistry of Products from the Radical Reaction of C_{60} with Azo(bisisobutyronitrile), *J. Org. Chem.* **1999**, *64*, 6257-6262.

Ford, W. T.; Nishioka, T.; McCleskey, S.; Mourey, T. H.; Kahol, P. Structure and Radical Mechanism of Formation of Copolymers of C_{60} with Styrene and with Methyl Methacrylate, *Macromolecules* **2000**, *33*, 2413-2423.

Ford, W. T.; Nishioka, T.; Qiu, F.; D'Souza, F.; Choi, J.-p., Dimethyl Azo(bisisobutyrate) and C_{60} Produce 1,4- and 1,16-Di(2-carbomethoxy-2-propyl)-1,x-dihydro[60]fullerenes, *J. Org. Chem.* **2000**, *65*, 5780-5784.
Ford, W. T.; Lary, A. L.; Mourey, T. H., Addition of Polystyryl Radicals from TEMPO-Terminated Polystyrene to C_{60}, *Macromolecules* **2001**, *34*, 5819-5826.

Other significant publications

Pan, Y.; Ford, W. T., Dendrimers with Both Hydrophilic and Hydrophobic Chains at Every End, *Macromolecules* **1999**, *32*, 5468-5470.
Miller, P. D.; Ford, W. T., A Survey of Alkyl Methacrylate Latexes As Aqueous Catalytic Media, *Langmuir* **2000**, *16*, 592-596.
Chen, Y.; Ford, W. T.; Materer, N. F.; Teeters, D., Facile Conversion of Colloidal Crystals to Porous Polymer Nets, *J. Am. Chem. Soc.* **2000**, *122*, 10472-10473.
Chen, Y.; Ford, W. T.; Materer, N.; Teeters, D. Conversion of Colloidal Crystals to Polymer Nets: Turning Latex Particles Inside Out, *Chem. Mater.* **2001**, *13*, 2697-2704.
Xu, Z.; Ford, W. T., Polystyrene Latexes Containing Poly(propyleneimine) Dendrimers, *Macromolecules* **2002**, *35*, 7662-7668.

Collaborators , current and last 4 years

B. Ackerson, N. Kotov, N. Materer, O. Spivey, P. Tong, and J. Wicksted, OSU
E. Amis and B. Bauer, NIST
L. Bokobza, Paris
R. Colby, Penn State
F. D'Souza, Wichita State
S. Foulger, Clemson
D. Guldi, Notre Dame
P. Kahol, Wichita State
W. Kutner, Polish Academy of Sciences
J. Mark, Cincinnati
T. Mourey, Eastman Kodak
M. Rubinstein, North Carolina
D. Teeters, Tulsa
S. Tolbert, UCLA
R. Vold, William & Mary
J. Zhu, Montreal

Ph.D. Advisor: The late D. J. Cram, UCLA
Postdoctoral Advisor: The late P. D. Bartlett, Harvard

Thesis Advisor and Postgraduate-Scholar Sponsor, last 5 years
J. Jethmalani, Calhoun Vision, Inc.
K. Hampton, Eastman Chemical Co.
R. Joseph, Cosmyl, Inc.
J. Kreider, Millikin Co.
A. Lary, Pharmacia, Inc.
P. Miller, U. of Wisconsin-La Crosse
T. Nishioka, Ibaraki, Japan
Y. Pan, Affymax Research Inst.
S. Pilcher, Northwestern Oklahoma State U.
E. Seabolt, PolyDyne Development Corp.
K. Vassilev, Bourgas, Bulgaria
Y. Chen, Schlumberger, Inc.